IS 7697 (1991): Phenyl Ethyl Methyl Ether [PCD 18: Natural and Synthetic Fragrance Materials]

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IS 7697 : 1991

(Reaffirmed 2002)

भारतीय मानक

फिनाइल इथाइल मिथाइल इथर — विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

PHENYL ETHYL METHYL ETHER — SPECIFICATION

(First Revision)

UDC 665·53 : 661·721

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

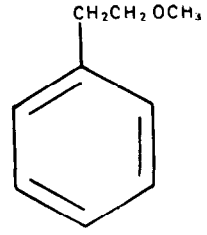
November 1991

Price Group 2

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Natural and Synthetic Perfumery Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Phenyl ethyl methyl ether, $C_9H_{12}O$ is prepared by the methylation of phenyl ethyl alcohol. It gives a green top note resembling the top note of *KEWDA* flower in which it occurs. It is represented by the following structural formula:



Molecular Mass 136

Methyl — β — phenyl ethyl ether

This standard was first published in 1975. At that time due to non-availability of standardized test method for chromatographic determination of this material, it was decided that GLC method would be included at a later date. This revision of this standard has been undertaken to include gas chromatographic method of analysis as the main method for determination of purity of phenyl ethyl methyl ether.

Besides, two new requirements namely minimum purity and peroxide value have been included and requirements for ester value and ester value after acetylation have been deleted from this revision.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

PHENYL ETHYL METHYL ETHER — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for phenyl ethyl methyl ether.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
326	Methods of sampling and test for natural and synthetic perfumery materials:
(Part 1) : 1984	Sampling (*second revision*)
(Part 2) : 1980	Preliminary examination of perfumery materials and samples (*second revision*)
(Part 3) : 1980	Relative density (*second revision*)
(Part 5) : 1986	Determination of refractive index (*second revision*)
(Part 6) : 1986	Determination of solubility in ethanol (*second revision*)
(Part 7) : 1980	Determination of acid value (*second revision*)
1070 : 1977	Water for general laboratory use (*second revision*)
2284 : 1988	Methods for olfactory assessment of natural and synthetic perfumery materials (*first revision*)
6597 : 1988	Glossary of terms relating to natural and synthetic perfumery materials (*first revision*)

3 TERMINOLOGY

For the purpose of this standard, the definitions of terms given in IS 6597 : 1988 shall apply.

4 REQUIREMENTS

4.1 The material shall be examined for its colour, clarity, separated water, and sediment as prescribed under IS 326 (Part 2) : 1980.

4.2 Solubility

When tested as prescribed under IS 326 (Part 6) : 1986, the material shall be clearly soluble in 4 volumes of 60 percent (*v/v*) ethyl alcohol.

4.3 The material shall also comply with the requirements given in Table 1.

5 PACKING AND MARKING

5.1 The materials shall be supplied in well closed containers, preferably glass, tin-lined, stainless steel or aluminium as agreed to between the purchaser and the supplier.

Table 1 Requirements for Phenyl Ethyl Methyl Ether

(*Clauses* 4.3 *and* 7.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Odour	Typical strong green note resembling *KEWDA* flower	IS 2284 : 1988
ii)	Relative density[1] at 27/27°C	0·941 0 to 0·947 0	IS 326 (Part 3) : 1980
iii)	Refractive index[2] at 27°C	1·494 0 to 1·497 0	IS 326 (Part 5) : 1986
iv)	Acid value, *Max*	0·5	IS 326 (Part 7) : 1980
v)	Peroxide value, *Max*	10	Annex A
vi)	Purity, percent by mass, *Min*	98	Annex B

[1]The correction factor for each degree Celsius change in temperature is 0·000 64.

[2]The correction factor for each degree Celsius change in temperature is 0·000 38.

5.2 The material shall be stored in a cool and dry place well protected from light.

5.2.1 The containers may also be marked with the Standard Mark.

6 SAMPLING

Representative samples of the material shall be drawn as prescribed in IS 326 (Part 1) : 1984.

7 TESTS

7.1 Tests shall be conducted as prescribed in col 4 of Table 1. Reference to the relevant standards is given in col 4 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (IS 1070 : 1977) shall be employed in tests.

NOTE— 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* (v)]

DETERMINATION OF PEROXIDE VALUE

A-1 REAGENTS

A-1.1 Acetic Acid — Analytical Reagent Grade.

A-1.2 Chloroform

A-1.3 Sodium Thiosulphate Solution — 0·01 N approximately.

A-1.4 Potassium Iodide — saturated solution.

A-1.5 Starch — freshly prepared saturated solution.

A-2 PROCEDURE

A-2.1 Weigh accurately about 2 g of the material into a conical flask (250 ml capacity) with provision for stoppering. Add 25 ml of acetic acid — chloroform mixture solution (3 · 2 *v/v*). Swirl to dissolve the material completely. Add 2 ml of freshly prepared saturated aqueous solution of potassium iodide. Allow the solution to stand with occasional shaking for 1 minute and then add 35 ml of distilled water. Add 2 ml of freshly prepared starch solution. Titrate against sodium thiosulphate (0·01 N). End point being the disappearance of the blue colour. A blank titration using all the reagents but without sample should also be carried out.

A-3 CALCULATION

A-3.1 Calculate the peroxide value in the material as follows:

$$\text{Peroxide value milliequivalents of peroxy-oxygen/kg} = \frac{(V_1 - V_2) \times N \times 1\,000}{M}$$

where

V_1 = volume in ml of sodium thiosulphate solution used for the test sample,

V_2 = volume in ml of sodium thiosulphate solution used for blank titration,

N = normality of sodium thiosulphate, and

M = mass of the sample in g.

ANNEX B

[*Table* 1, *Sl No.* (vi)]

GAS CHROMATOGRAPHIC ANALYSIS OF PHENYL ETHYL METHYL ETHER

B-0 GENERAL

B-0.1 The chromatographic conditions given here are for guidance only.

B-0.2 Outline of the Method

A sample of the material is dissolved in a suitable solvent (namely, hexane cyclohexane and petroleum ether) and is injected into the gas chromatograph from where it is carried by the carrier gas from one end of the column to the other. During its movement, the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after

another and are detected by suitable means whose response is related to the amount of a specific component leaving the column.

B-1 APPARATUS

Any gas chromatograph capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. The typical chromatogram for phenyl ethyl methyl ether using a chromatograph with the following chromatographic conditions is shown in Fig. 1:

Sample	: Phenyl Ethyl Methyl Ether
Column	
Material	: Stainless steel
Length	: 2 m
OD	: 0·32 cm
ID	: 0·20 cm
Stationary phase	: FFAP[1], 10 percent by mass
Solid support	: Chromosorb WAP
Carrier Gas	: Nitrogen
Conditions	
Column temperature	: 150°C
Injection port temperature	: 250°C
Detector	
Type	: FID
Temperature	: 250°C

[1] Free fatty acid phase (FFAP) in carbowax 20 m treated with nitrophthalic acid.

B-2 CALCULATION

B-2.1 Area Measurement (*See* Note 1)

Since normal peaks approximate a triangle, the area is measured by multiplying the peak height times the width of half-height. The normal peak base is not taken since large deviations may be observed due to tailing or adsorption. This technique is rapid, simple and fairly accurate when peaks are symmetrical and of reasonable width.

B-2.2 Area Normalization (*See* Note 2)

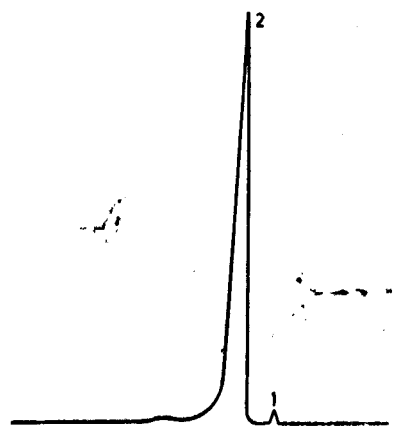
By normalizing, it is meant, calculating the percentage composition by measuring the area of each and dividing the individual areas by total area, for example,

$$\text{Percentage of } A \times \frac{\text{Area of } A}{\text{Total Area}} \times 100$$

NOTES

1 Other methods of area measurements, namely, triangulation, disc integrator and electronic digital integrator, if fixed with GLC machine, would be of great advantage.

2 Internal standardization may be used if pure appropriate internal standard is available. This method is known as relative or indirect calibration,



1—Solvent, 2--PEME

FIG. 1 TYPICAL CHROMATOGRAM OF PHENYL ETHYL METHYL ETHER

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